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Materials & Mechanics Department
June 28, 1990

Dr. Steven G. Fishman
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800 North Quincy
Arlington, VA 22217

Subject: SwRI Project 06-8602
"Micromechanisms of Fatigue Crack Growth and Fracture Toughness in Metal Matrix Composites"
Contract No. N00014-85-C-0206
FINAL REPORT

Dear Dr. Fishman:

Enclosed is the final report of the above referenced contract. If you have any questions or comments, please feel free to contact me (512/522-2314).

Very truly yours,

David L. Davidson
Project Manager

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Enclosure

cc: G. R. Leverant
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B. J. Andrews

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Gerald R. Leverant, Director
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N00014-85-C-0206
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MICROMECHANISMS OF FATIGUE CRACK GROWTH AND FRACTURE TOUGHNESS IN METAL MATRIX COMPOSITES

FINAL REPORT
SwRI Project No. 06-8602

Prepared For
Office of Naval Research
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Arlington, VA 22217

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June 1990

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Research objectives under which this work was initiated were as follows: "To understand the mechanisms of subcritical crack extension and fracture toughness in particulate-reinforced metal matrix composites projected for Naval applications in which one or more of these parameters control service." The factors thought to be important in 1985 were the processing variables by which these materials were fabricated and the role of SiC particles in altering fatigue crack growth and fracture toughness of the matrix alloys.

The methods used in reaching for these objectives include the techniques of experimental micromechanics. Experiments in fatigue crack growth and fast fracture were conducted for a variety of composites obtained from different manufacturers, and these materials were thoroughly characterized. Materials characterized were picked in order to: 1) determine the effects of differences in matrix composition and heat treatment, 2) determine the effect of SiC volume fraction, and 3) account for differences in particle size. Materials were obtained from two different manufacturers. IN-9051+15v/o SiC (essentially Al-4Mg) and IN-9021+15v/o SiC, both made by mechanically alloying and powder metallurgy were obtained from Novamet. The remainder of the composites made by ingot metallurgy by Dural Aluminum Composites Co., were: Al-4Mg+15v/o SiC, 2014+15v/o SiC, 2014+25v/o SiC, 2024+15v/o SiC, 2014+15v/o SiC, and 7475+15v/o SiC.

After a detailed investigation, the fracture characteristics of these composites were found to be controlled by two factors: 1) ductility of the matrix, and 2) the limitations on slip imposed by the SiC. So far as could be measured, fracture toughness and the fatigue threshold were not controlled by crack bridging, crack trapping at particles, microcracking, or the cracking of SiC particles. Microvoid formation was found to be negligible.

Questions about crack growth in ceramics and the availability of material led to a relatively small study of fracture in partially stabilized zirconia. At ambient temperature, this ceramic exhibited a threshold for fatigue crack growth, crack growth was discontinuous, and cracks advanced through the formation and breakdown of a slip line; striations were found on the fracture surface. At elevated temperature, stable fatigue crack growth was almost non-existent. It was possible to account for the fracture toughness by computing the work done in forming the "plastic" zone at fast fracture from the measured strain distribution.

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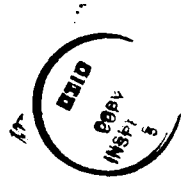

I. RESEARCH OBJECTIVES AND METHODOLOGY

Research objectives under which this work was initiated were worded as follows: "The program outlined here is aimed at addressing both subcritical crack extension and fracture toughness in particulate-reinforced metal matrix composites projected for Naval applications in which one or more of these parameters control service." The factors thought to be important at that time were the processing variables by which these materials were fabricated and understanding just how SiC particles altered fatigue crack growth and fracture toughness.

The methodology proposed was a detailed examination of fracture using the techniques of experimental micromechanics, and from information gained in this way, to determine the mechanisms controlling fracture of these materials. Experiments in fatigue crack growth and fast fracture would be conducted for a variety of composites obtained from different manufacturers, and these materials would be thoroughly characterized.

II. MATERIALS AND EXPERIMENTAL TECHNIQUES

Materials chosen for characterization were picked in order to: (1) compare differences in matrix composition and heat treatment, (2) compare the effect of SiC volume fraction, and (3) compare differences in particle size. To get materials with all these characteristics necessitated acquisition of material from two different manufacturers. Novamet supplied, without cost, IN-9052+15v/o SiC (essentially Al-4Mg) and IN-9021+15v/o SiC, both made by mechanically alloying followed by powder consolidation processing. These composites had particle sizes much smaller than could be obtained by other manufacturing processes. The remainder of the composites evaluated were purchased from Dural Aluminum Composites Co. These composites were made by introducing SiC particles into the molten metal, which was then cast and extruded. The combinations of matrix alloys and SiC characterized were:

Al-4Mg+15v/o SiC,
2014+15v/o SiC,
2014+25v/o SiC,
2024+15v/o SiC,
2014+15v/o SiC,
7475+15v/o SiC.

Al-4Mg+15v/o SiC was used to compare directly with IN-9052+15v/o SiC. Neither of these matrices was heat treatable. Composites with the 2000 and 7000 series matrices were evaluated in both as-received and peak-strength ageing conditions. For comparison, ingot produced, unreinforced 2024-T351 was evaluated also.

Characterization of Microstructures was accomplished with great expenditure of time and effort. Light microscopy, scanning electron microscopy, and transmission electron microscopy were all used. The large spread in particle size, especially in the INCO composites, required integration of all the methods used for particle sizing. Because of the level of difficulty, only two of the composites were fully characterized: IN-9052+15v/o SiC and 2014+15v/o SiC. The first material exhibited a bimodal distribution of particles, with peak concentrations of 5% at 0.3 μm and 17% at 9 μm [1]. The ingot derived composite, conversely, had a peak concentration of a little over 5% at 6.6 μm. The particle size distribution for this material was described by a log-normal function [2].

Fatigue and fracture toughness were characterized using several testing machines: a computer controlled laboratory fatigue machine was used to screen the large number of alloys and heat treatments. Fatigue cracks were initiated in compact specimens and down loaded according to the ASTM standard to obtain a threshold value, ΔK_{th}, then up loaded to obtain a fatigue crack growth rate curve. The stress intensity factor was determined at the point of fast fracture, and this value was used as a measure of the fracture toughness K_c. The material exhibiting the lowest K_c was IN-9052+15v/o SiC, and the material exhibiting the largest K_c was 2014-PA+15v/o SiC. The parameters describing fatigue crack growth and measured values of K_c for all the materials tested were tabulated in ref. [3].

The two materials exhibiting the largest and smallest K_c were evaluated in detail using the loading stage for the SEM and a laboratory closed loop fatigue machine manually controlled. Crack length was measured by traveling microscope. The results obtained with this equipment largely agreed with those obtained using the computer controlled equipment. However, two K_c tests were conducted within the SEM for 2014-PA+15v/o SiC because of the large spread found (18 to 28 $MPa\sqrt{m}$) [4].

The stereoimaging technique was used to make measurements of displacements around crack tips, both under fatigue loading and at the point of fast fracture. From these displacements, crack opening displacements and strains were determined. Strain distributions and crack driving forces were determined with this information. Thus, a number of phenomena associated with fracture were quantitatively examined.

III. SUMMARY OF RESULTS

Fatigue crack growth rates were characterized with the relation:

$$da/dN = B\Delta K^s \tag{1}$$

and a value of ΔK_{th} was measured for each composite [5], allowing eq. (1) to be rewritten as

$$da/dN = B'\Delta K_{eff}^{s'} \tag{2}$$

where $\Delta K_{eff} = \Delta K - \Delta K_{th}$. Correlations were found between the coefficients B and s in eq. (1), as they have for other materials, and a correlation was found between ΔK_{th} and s. A simple model based on the size and volume fraction of SiC particles was used to compute ΔK_{th}. The work of investigators for similar composites was included in these correlations. Therefore, by knowing the microstructural characteristics of a new (untested) composite, it should be possible to estimate the fatigue threshold and crack growth rate curve solely on the basis of microstructure. One value of B' and s' in eq. (2) may be used to describe this entire family of composites.

Fracture surface roughness has been correlated with the magnitude of fracture toughness for steels, so a similar link was sought for these composites. There has also been speculation about a correlation between ΔK_{th} and fracture surface roughness. Fracture surface roughness was measured for fracture surfaces generated by both fatigue and fast fracture [3]. Roughness was found to be described very well by the fractal dimension for both cases. However, no correlation was found between fracture surface roughness and either ΔK_{th} or K_c for these composites. Upon further investigation, the reasons for this result became more apparent, in that the mechanisms by which crack growth is occurring in these materials is influenced in only a minor way by the resulting crack path.

Since these tests were conducted in humid air, some environmental effects might have influenced these results. However, a strong effect of water vapor on ΔK_{th} was proved to be unlikely, since no excessive build up of oxides was detected during fractography. Breakage of SiC particles has also been raised as an issue with these materials, but only the larger particles were found to break, and this mechanism was not determined to be much of a factor in controlling fracture, either fatigue or fracture toughness. One reason for fracture of the large SiC, at least for the case of the mechanically alloyed composite, was that these particles were often cracked during processing [1].

Fractography also indicated the formation of very few fatigue striations. Dynamic observations in the SEM of fatigue crack growth indicated that cracks were growing through these materials as though no SiC were present; e.g., it still required a number of cycles for the crack to lengthen when the stress intensity factor was in the near-threshold region. Thus, cracks were still growing and arresting even though few striations were found to evidence this behavior.

Virtually no microvoids were found in the fast fracture regions of either of the composites carefully examined, indicating that little energy was being absorbed by microvoid growth and coalescence. However, tear ridges were found - these may be viewed as large, disconnected "microvoids" which are unorganized, but the main point is that tear ridges cover less area than the regions of large plasticity which surround an extensive, well developed microvoid system; thus, the energy expended in forming these tear ridges was low. The energy of microvoid formation was estimated by a model developed in [1] and found to be at least a factor of 10 less than that expended in forming the plastic zone of the crack tip. Surprisingly, no other model for energy expenditure during void formation can be found in the literature.

IV. DETAILED STUDIES OF THE LOWEST AND HIGHEST FRACTURE TOUGHNESS MATERIALS

The mechanically alloyed IN-9052 matrix composite was found to have the lowest fracture toughness. The physical characteristics of this material may be summarized as follows [1]: strong matrix particle interfaces; bimodal particle distribution, but with a fairly good particle dispersion; a large volume fraction of Al_4C_3, Al_2O_3, and MgO particles; and a subgrain size of about 0.67 μm. The material exhibited a fairly large porosity. Fatigue crack growth was by intermittent crack advance, but only a few striations were found, and those were very closely spaced (0.055 μm); fatigue cracks seemed to be attracted to pores. Fast fracture occurred with the formation of few dimples, but a few large broken SiC particles (probably precracked) were found. At the point of fast fracture, strain distribution was measured and a model for computing the work expended in forming the plastic zone was derived. This plastic work term, W_p, was related to K_c through the modified Griffith equation: $K_c = \sqrt{(W_p E)}$, and through this process it was found that the stress intensity at fast fracture could be accounted for by the plastic work done during fracture.

This modeling effort has lead to a method for examining the mechanisms of fracture for several materials. By determining the plastic work expended in fast fracture, an assessment of the effectiveness of other proposed mechanisms of fracture may be examined; e.g., crack bridging, fracture of SiC particles, and microcracking ahead of the main crack tip.

The ingot produced 2014 matrix composite was found to have the highest fracture toughness, although quite a range in toughnesses was measured during repeated tests (18 to 28 MPa$\sqrt{m}$). The physical characteristics of this material may be summarized as follows [2]: strong matrix particle interfaces, particle distribution with peak at 6.6 μm, and with a fairly poor particle dispersion; many clumps of SiC were found. Some interface separation occurred within clumps, presumably due to insufficient working which breaks up oxides at the matrix-particle interfaces and allows adhesion to occur. There was also 0.03 volume fraction of intermetallics, mostly containing copper, some as large as 20μm in size. No quantitative method was found for characterizing the clumping in this material.

Tensile tests were performed in the SEM [4] and it was found that particle clumping had a large effect on the magnitude of strains within the matrix, and that these were found to greatly exceed the overall elongation measured for the specimens.

Fatigue crack growth was by intermittent crack advance, and very few striations were found [2]. At some levels of ΔK, microcracks formed near the main crack, and it was possible through analysis to show that these had very little effect on the stress intensity of the main crack. Measured strain distributions were found to be significantly altered by the presence of SiC particles, particularly at low ΔK [2]. This finding fit well with the concept that the threshold was controlled by SiC limited slip in the matrix.

Just as for the mechanically alloyed composite, fast fracture occurred with the formation of few dimples, and a few large broken SiC (perhaps precracked) also were found [4]. At the point of fast fracture, strain distribution was measured and the previously developed model for computing the work expended in forming the plastic zone was used. Again, it was found that the stress intensity at fast fracture could be accounted for by the plastic work done during fracture. Crack bridging, which occurred during one of the fast fracture experiments, was found to be completely ineffective [4].

In summary, it appears as though the fracture characteristics of these composites is controlled by two factors: (1) the ductility of the matrix, and (2) the limitations on slip imposed by the SiC. So far as could be measured, fracture toughness and the fatigue threshold were not controlled by crack bridging, crack trapping at particles, microcracking, or the cracking of SiC particles. The general concepts controlling fracture toughness which have come from this work were summarized in the chapter [6] of a book on metal matrix composites.

V. FATIGUE AND FAST FRACTURE OF PARTIALLY STABILIZED ZIRCONIA

The confusion amongst ceramists about fatigue crack growth in ceramics led to a relatively small study of fracture in partially stabilized zirconia [7]. The material used was a gift of Dr. Mike Swain from CSIRO in Australia, and it was evaluated because it has been extensively characterized. At ambient temperature, this ceramic (or composite) acts very much like a metal, in that it had a threshold for fatigue crack growth, crack growth was discontinuous at low ΔK, and cracks were observed to lengthen through the formation and breakdown of what appeared to be a slip line, the same mechanism as found for aluminum alloys. Periodic crack arrest markings (striations) were found on the fracture surface, although they had a different appearance than for metallic alloys. A previously developed model for fatigue crack growth was used to derive the low cycle fatigue and stress-failure (S-N) curves, and a good comparison was made with measured S-N curves. For metals, the slope of the crack growth rate curve with increasing ΔK is 2-6, but for this PSZ, the slope was 25.

At elevated temperature, the range of ΔK over which stable fatigue crack growth occurred was decreased and the slope of the fatigue crack growth rate curve was increased. The higher the temperature, the smaller the range of stable fatigue crack growth and the lower the fracture toughness; extrapolating tests at 450 and 650°C indicated that by 700-750°C, no stable region of crack growth would be expected, with a K_c of only about 3 MPa$\sqrt{m}$. It was possible also for this material to account for the level of K_c measured by computing the work done in forming the "plastic" zone at fast fracture from the measured strain distribution.

The mechanism responsible for the apparent plasticity exhibited by this material is transformation of monoclinic zirconia to tetragonal, rather than the generation and motion of dislocations as is the case for the aluminum alloy composites studied. Although PSZ is classified as a ceramic, it is certainly unique amongst ceramics, and it is doubtful that the fracture characteristics of this material resemble those of the more typical ceramics, such as the oxides.

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